

# इंटरनेट

# मानक

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“Step Out From the Old to the New”

IS 12107-10 (2001): Methods of Chemical Analysis of Alumino-silicate Refractory Materials, Part 10: Determination of Iron Manganese, Calcium and Magnesium by Atomic Absorption Spectrometric Method [MTD 13: Ores and Raw Materials]



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“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक

एलुमिनो-सिलिकेट अग्नि सह सामग्रियों के  
रासायनिक विश्लेषण की प्रणाली

भाग 10 परमाणु अवशोषण स्पेक्ट्रोमेट्रिक विधि द्वारा  
लोह, मैंगनीज, कैल्सियम एवं मैग्नीसियम का निर्धारण

*Indian Standard*

METHODS OF CHEMICAL ANALYSIS OF  
ALUMINO-SILICATE REFRACTORY MATERIALS

PART 10 DETERMINATION OF IRON MANGANESE, CALCIUM AND MAGNESIUM  
BY ATOMIC ABSORPTION SPECTROMETRIC METHOD

ICS 71.040.50; 73.060.01

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## FOREWORD

This Indian Standard (Part 10) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industry Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Alumino-silicate refractory materials containing alumina ( $\text{Al}_2\text{O}_3$ ) and silica ( $\text{SiO}_2$ ) in varying proportions is made synthetically by heating aluminium trifluoride at 1 000 - 1 200°C with silica and water vapour. It is used in kilns, laddles and furnaces that operate at a high temperature or under the conditions for which fireclay refractories are not suitable.

The analysis of its constituents is covered in various parts of this standard. This part covers the determination of iron, manganese, calcium and magnesium by atomic absorption spectrometric method. The other parts of the standard are as follows:

- Part 1 Determination of loss on ignition
- Part 2 Determination of silica
- Part 3 Determination of aluminium
- Part 4 Determination of phosphorus
- Part 5 Determination of titanium
- Part 6 Determination of iron
- Part 7 Determination of manganese
- Part 8 Determination of calcium and magnesium
- Part 9 Determination of sodium and potassium

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical value (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## *Indian Standard*

# METHODS OF CHEMICAL ANALYSIS OF ALUMINO-SILICATE REFRACTORY MATERIALS

## PART 10 DETERMINATION OF IRON MANGANESE, CALCIUM AND MAGNESIUM BY ATOMIC ABSORPTION SPECTROMETRIC METHOD

### 1 SCOPE

This standard (Part 10) describes the determination of iron, manganese, calcium and magnesium in alumino-silicate refractory materials by atomic absorption spectrometric method in the ranges given below:

<i>Element</i>	<i>Percent by Mass</i>
Iron	0.2 – 2.0
Manganese	0.02 – 0.12
Calcium	0.01 – 0.10
Magnesium	0.05 – 0.50

### 2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid ( <i>second revision</i> )
460	Test sieves: Part 1 Wire cloth test sieves ( <i>third revision</i> )
(Part 1) : 1985	
1070 : 1992	Reagent grade water ( <i>third revision</i> )
1528	Methods of sampling and physical tests for refractory materials: Part 7
(Part 7) : 1974	Methods of sampling and criteria for conformity ( <i>first revision</i> )

### 3 SAMPLING AND PREPARATION OF TEST SAMPLE

**3.1** The sample shall be drawn in accordance with the procedure laid down in IS 1528 (Part 7).

#### 3.2 Laboratory Sample

The sample shall be crushed in any type of crusher with hardened tool steel faces, with due precautions to prevent contamination of sample with steel particles from the sampling equipment during crushing and grinding. After quartering, 20 g of sample should be carefully screened with magnet to remove extraneous

iron particles. It shall then be ground in agate mortar and pestle so that it passes completely through IS sieve 15 (aperture 75  $\mu\text{m}$ ) [see IS 460 (Part 1)].

#### 3.3 Test Sample

It is prepared for the purpose of chemical analysis by drying sufficient quantity of laboratory sample (see 3.2) in a glass weighing bottle, in an air oven at 105-110°C.

### 4 QUALITY OF REAGENTS

Unless specified otherwise, reagent grade water (see IS 1070) shall be employed for the test.

### 5 DETERMINATION OF IRON, MANGANESE, CALCIUM AND MAGNESIUM

#### 5.1 Outline of the Method

Sample is fused with a mixture of sodium carbonate and sodium borate and dissolved in nitric acid. Suitable aliquot after removal of boron and silica are aspirated into the flame atomic absorption spectrophotometer and the absorbance is measured at respective wavelength.

#### 5.2 Reagents

**5.2.1** Sodium carbonate, anhydrous.

**5.2.2** Sodium borate, solid.

**5.2.3** Concentrated Nitric Acid, r.d = 1.42 (conforming to IS 264).

**5.2.4** Hydrogen Peroxide, 30 percent (v/v).

**5.2.5** Hydrofluoric Acid, 40 percent (v/v).

**5.2.6** Dilute Sulphuric Acid, 1:1(v/v).

**5.2.7** Potassium Bisulphate, Solid.

**5.2.8** Methyl Alcohol

**5.2.9** Dilute Nitric Acid, 1:4 (v/v).

**5.2.10** Lanthanum solution (1ml = 50mg La) — Weigh to the nearest 0.10g, 15.6 g of lanthanum nitrate hexahydrate  $[\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}]$  and dissolve in water. Make up volume to 100 ml and mix.

**5.2.11** Standard Iron Solution (1 ml = 1 mg Fe) —

Dissolve 1.000 g of electrolytic iron in 20 ml of dilute nitric acid and dilute to 1 000 ml with water, maintaining the acidity of 3 percent (v/v) nitric acid and mix.

On further stepwise dilution obtain working standard of 1 ml=10 µg Fe.

#### 5.2.12 *Standard Manganese Solution* (1 ml =1 mg Mn).

Dissolve 1.000g of manganese (spec-pure) strip in a minimum volume of dilute nitric acid (1:1) and dilute to one litre, maintaining acid concentration of 3 percent (v/v) nitric acid and mix.

On further stepwise dilution obtain working standard solution of 1 ml = 50 µg Mn.

**5.2.13 *Standard Calcium Solution*** (1 ml =1 mg Ca) — Weigh 2.497 2 g calcium carbonate, previously dried for 2 hours at 110°C and cooled in a desiccator. Transfer to a 250 ml beaker and dissolve in 10 ml of dilute nitric acid (1 : 1) and make up the volume to one litre with water, maintaining the overall acid concentration of nitric acid to 3 percent (v/v) and mix.

On further stepwise dilution, obtain the working standard solution of calcium to give 1 ml =25 µg.

**5.2.14 *Standard Magnesium Solution*** (1 ml = 1 mg Mg) — Dissolve 1.000 g of magnesium metal in 20 ml of dilute nitric acid and dilute to one litre with water, maintaining acid concentration of dilute nitric acid 3 percent (v/v) and mix.

Prepare suitable dilute solution, by stepwise dilution to give 1 ml =25 µg Mg.

**5.2.15 *Aluminium Background Solution*** (1 ml = 20 mg Al) — Dissolve 20 g of aluminium shavings/turnings in 200 ml of sodium hydroxide solution (5 N). When aluminium has dissolved, add 400 ml of nitric acid. Make upto one litre in volumetric flask with water and mix.

### 5.3 Procedure

#### 5.3.1 *Test Portion*

Weigh, 0.50 g of the sample. Transfer to a platinum crucible.

#### 5.3.2 *Decomposition of the Test Portion*

Add 5 g of fusion mixture (1:1 mixture of sodium carbonate and sodium borate). Mix well with a glass rod or by swirling slowly. Cover the crucible. Heat slowly on a burner at low flame, and then increase the temperature and keep it for half an hour with occasional swirling of melt. Cool and observe that a transparent glass is obtained, so as to ensure complete dissolution of the sample. Cool it.

Alternatively the samples may be decomposed by  $\text{HNO}_3$ -HF-HClO<sub>4</sub> in teflon beaker and heat on a hot plate until dense fumes of perchloric acid are liberated. Cool, dissolve the mass in 10 ml (1:1) HCl make upto the mark after adding releasing agent 1 percent La or Sr salt for the determination of Ca and Mg. For Fe and Mn, no releasing agent is required to be added.

It will save time and labour to remove boron and silica. Use of Na<sub>2</sub>CO<sub>3</sub> and sodium borate would lead to encrustation on the burner head which would require cleaning quite often.

**5.3.3** Dissolve the contents in nitric acid and transfer to 250 ml beaker and boil. Filter, if necessary, washing the residue with warm water. Preserve the filtrate (A). Dry the filter paper alongwith residue in the same crucible. Char and ignite at 600°C till the whole carbon is completely destroyed. Cool and moisten with few drops of water. Add 10 ml hydrofluoric acid and 1 ml of dilute sulphuric acid (1 : 1). Slowly evaporate off on a steam bath and then heat to fume. Bring the left over residue if any, into the solution by fusion with a pinch of potassium bisulphate and bring into solution with nitric acid and water. Mix it with filtrate (A). Make up the volume to 100 ml in volumetric flask.

**5.3.4** Take 25 ml aliquot of the solution (see 5.3.3) in a platinum dish of 100 ml capacity. Add 10 ml of methyl alcohol and evaporate cautiously on steam bath. Repeat methyl alcohol treatment thrice, while washing the sides of the dish with water. Add 10 ml of hydrofluoric acid and 2 ml of nitric acid. Evaporate to dryness on steam bath. Repeat treatment thrice and finally take the dried mass in solution with 5 ml of dilute nitric acid (1 : 4) and water. Transfer into a 25 ml volumetric flask. Add 2.5 ml of lanthanum solution. Make up to volume and mix well.

#### 5.3.5 *Preparation of Calibration and Blank Solution*

**5.3.5.1** Take seven numbers 25 ml volumetric flasks and to each flask add 2.5 ml of lanthanum solution (see 5.2.10) 2 ml of aluminium solution (see 5.2.15) and the standard solutions as per Table 1. Dilute to the mark and mix well. The zero number of the series will serve as the reference solution.

**5.3.5.2** Prepare a blank solution using same quantities of all the reagents but without the sample and following the procedure specified in 5.3.2 to 5.3.4.

#### 5.3.6 *Adjustment of the Atomic Absorption Spectrometer*

Follow the instructions of the manufacturer in preparing the instrument. Switch on the instrument and the appropriate hollow cathode lamp. Fit the burner with either air-acetylene (for iron and manganese), or nitrous oxide-acetylene (for calcium

**Table 1 Standard Solutions**  
(Clause 5.3.5.1)

Standard Solution µg/ml				Corresponding Mass, mg in 100 ml				Percent Metal in Refractory on Basis of 0.5 g/Sample in 100 ml			
Fe (1) O <sup>1)</sup>	Mn (2) O <sup>1)</sup>	Ca (3) O <sup>1)</sup>	Mg (4) O <sup>1)</sup>	Fe (5) O <sup>1)</sup>	Mn (6) O <sup>1)</sup>	Ca (7) O <sup>1)</sup>	Mg (8) O <sup>1)</sup>	Fe (9) O <sup>1)</sup>	Mn (10) O <sup>1)</sup>	Ca (11) O <sup>1)</sup>	Mg (12) O <sup>1)</sup>
10	1	0.5	2.5	1	0.1	0.05	0.25	0.2	0.02	0.01	0.05
20	2	1.0	5.0	2	0.2	0.10	0.50	0.4	0.04	0.02	0.10
40	3	2.0	10.0	4	0.3	0.20	1.0	0.8	0.06	0.04	0.20
60	4	3.0	15.0	6	0.4	0.30	1.5	1.2	0.08	0.06	0.30
80	5	4.0	20.0	8	0.5	0.40	2.0	1.6	0.10	0.08	0.50
100	6	5.0	25.0	10	0.6	0.50	2.5	2.0	0.12	0.10	0.50

<sup>1)</sup> Process blank.

and magnesium) and light the appropriate flame. Wait for about 20 minutes for stabilization. Set the wavelength at 386 nm for iron, 279.5 nm for manganese, 422.7 nm for calcium and 202.5 nm for magnesium. Optimize instrument response by adjusting the wavelength, fuel, air, burner and nebulizer while aspirating the highest calibration solution. As the sensitivity may vary from instrument to instrument the concentration of the standard series and of the test solution should be adjusted accordingly. At the same time check the linearity of the calibration curve.

Aspirate water and one of the calibration solution repeatedly to ensure that there is no drift of absorbance. Finally aspirate water and set the absorbance to zero reading.

### 5.3.7 Atomic Absorption Measurement

**5.3.7.1** Aspirate first the blank solution and then the calibration solution in increasing order of concentration, aspirating water in between each aspiration of the solution and record the absorbance reading. Then aspirate the test sample and note the absorbance. Each aspiration should be made at least three times and take the average value. Solids which build up on the burner slit must be removed, otherwise

they will cause a decrease of sensitivity.

**5.3.7.2** Deduct the blank value from the absorbance of the standard as well as the test solution.

**5.3.7.3** Prepare a calibration curve by plotting the absorption value of the standard solutions against the concentration in µg/ml of the element

**5.3.7.4** Compute the concentration of the test solution (corrected for the blank) to µg of the element per ml from the calibration curve.

### 5.4 Calculations

The analyte concentration, as percentage of mass is calculated from the following relation:

$$A, \text{ percent by mass} = \frac{m}{M} \times 100$$

where

$A$  = Analyte element,

$m$  = Mass in microgram of analyte/ml of test solution obtained from calibration graph, and

$M$  = Sample concentration in µg/ml in test solution.



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